

## Article

# Compatibility of Automatic Transmission Fluids with Structural Polymers Used in Electrified Transmissions

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**Featured Application:** Electric vehicle transmissions.

**Abstract:** The use of the electric motor (EM) inside the transmission in electric vehicles (EVs) requires compatibility between the automatic transmission fluids (ATFs) and the materials of the EM and the transmission. The goal of this work is to study the compatibility of four conventional ATFs with three structural polymers (PEEK, PTFE, and PA66) and the feasibility of using them in EVs with the EM located inside the transmission. Changes in volume, hardness, tensile strength, and elongation at break were determined in the polymers after ageing in the ATFs. The polymers were aged in each ATF at 100 °C for periods of time of 168, 336, 504, and 672 h. Complementary tests such as XRD, FT-IR, and DSC were performed to explain the changes found in the measured mechanical properties. PEEK and PTFE showed very low variations in their mechanical properties, while PA66 showed significant changes in elongation at break. The XRD, FT-IR, and DSC tests revealed a change in the crystalline structure of PEEK and PA66. The FT-IR results showed that the polymers were affected more by the time of ageing than by the type of ATF. All of the ATFs showed high compatibility with PEEK and PTFE, and medium compatibility with PA66.

**Keywords:** electric vehicles; automatic transmission fluids; materials compatibility; polymers



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## 1. Introduction

The electric vehicle (EV) has become the main solution in the struggle to minimise transportation-related emissions, which represent 14% of the total weight of greenhouse gasses that are emitted, according to the Intergovernmental Panel on Climate Change [1]. EVs have an average efficiency of 77% compared to 21% for vehicles driven by an internal combustion engine (ICE) [2]. The greater efficiency of EVs results in a large reduction in CO<sub>2</sub> emissions [3], especially when using electricity generated from renewable sources [4].

There are different classes of EVs, including battery electric vehicles (BEV), extended range electric vehicles (EREV), hybrid electric vehicles (HEV), plug-in hybrid electric vehicles (PHEV), mild hybrid electric vehicles (MHEV), and fuel cell electric vehicles (FCEV). Regardless of the EV type, the EM can be placed in different positions [5]. The position of the EM with respect to the ICE has an important role in the design and operation of hybrid vehicles. Although there is no industry agreement, the position of the EM is usually referred to as P0, P1, P2, P3, or P4 [6]. P0 and P1 are used when the EM is located before or after the ICE, respectively, and outside the transmission. In these cases, little change is expected for the ATFs because they do not interact with the EM. P2 and P3 refer to the case of an EM inside the transmission housing, either immediately before or immediately after the transmission, so the EM is in contact with the ATF. P4 describes the

arrangement in which the EM drives the rear axle [6], and hence is physically separated from the transmission and ICE.

When the EM is located within the transmission of the hybrid vehicle, the automatic transmission fluids (ATFs) come into contact with the EM, so fluid formulators must bear the following several requirements in mind: thermal compatibility, magnetic compatibility, corrosion protection, the problem of aeration and foaming at high speeds, electrical compatibility, and material compatibility [7–9]. To date, EVs are still using conventional ATFs to lubricate electrified drivetrains, and they have not been optimized for this application [10,11]. A variety of polymers are used in the electric motors, mainly for structural purposes, such as, for example, plastic bearing housings, and the ATFs that are employed must be compatible with these polymeric components. Among these polymers can be found polyether ether ketone, polyamide (commonly known as nylon), polyethylene, and polytetrafluoroethylene, but new materials that can withstand higher loads and operation temperatures will begin to be used [6,8].

Haverly et al. [12] studied the compatibility of some structural polymers (polyether ether ketone-PEEK, polytetrafluoroethylene-PTFE, and ultrahigh-molecular-weight polyethylene-UHMW PE) with different bio-oils. They found that PEEK and PTFE showed negligible degradation after ageing. Lann et al. [13] and Decker et al. [14] studied the compatibility of PA66 with ATFs. Lann et al. [13] pointed out the tendency of PA66 to crystallize when aged in ATFs, although they concluded that it was the high temperature that caused this effect. They also reported a drop in tensile strength. Decker et al. [14] confirmed the crystallization of PA66 in ATF after 2000 h at 130 °C, although they specified that the degradation was even greater in air at the same temperature, due to thermo-oxidation. Nonetheless, EV manufacturers recommend the use of conventional ATFs (originally designed for vehicles driven by ICEs) in EVs, without any reference to their overall compatibility when the EM is located inside the transmission.

The objective of this research is to study the compatibility of four conventional ATFs with three structural polymers and the feasibility to using them in EVs with the EM located inside the transmission. Changes in volume, hardness, tensile strength, and elongation at break were determined for the polymers (polytetrafluoroethylene—PTFE, Polyamide 66—PA66, and polyether ether ketone—PEEK), taking into account the recommendations provided by international standards [15]. The tested “material/oil” combinations were aged for 168 h as specified in the standards and, in addition, for longer time periods (336, 504, and 672 h). Additionally, X-ray diffraction (XRD), infrared spectroscopy (FTIR) and differential scanning calorimetry (DSC) tests were also performed on the polymers in order to explain the changes in their mechanical properties.

## 2. Materials and Methods

### 2.1. Materials

Four conventional automatic transmission fluids (ATFs), commonly used by numerous car manufacturers in their current models, were selected for the material compatibility tests. The composition and main properties of the ATFs are shown in Tables 1 and 2.

**Table 1.** Composition of the ATFs.

ATF	Base Oils (wt.%)	Additive Package (wt.%)
Oil A	Group I, 4.9 cSt/100 °C (77.7) Group I, 2.7 cSt/100 °C (10.6)	11.7
Oil B	Group III, 3.0 cSt/100 °C (38.3) Group III, 6.5 cSt/100 °C (50.7)	11.0
Oil C	Group III, 3.0 cSt/100 °C (45.0) Group III, 6.5 cSt/100 °C (38.5)	17.0
Oil D	Group III, 3.0 cSt/100 °C (36.5) Group III, 4.2 cSt/100 °C (36.5)	27.0

**Table 2.** Properties of the ATFs.

Properties	Oil A	Oil B	Oil C	Oil D
Density, 15 °C [g/cm <sup>3</sup> ]	0.869	0.847	0.854	0.854
Viscosity, 100 °C [cSt]	7.8	6.0	7.2	6.9
Viscosity, 40 °C [cSt]	43	30	35	34
Viscosity Index	161	151	181	165

Three different polymers were used in the compatibility tests. These materials (PEEK, PTFE, and PA66) are structural polymers commonly used in electric vehicles and, in some specific configurations, could be in contact with the transmission lubricants. The characteristics of the different materials are shown in Table 3. The specimens used in the compatibility tests were extracted from sheets of each material with the dimensions specified (type V) in the ASTM D638 standard [6,8].

**Table 3.** Polymer properties.

Properties	PEEK <sup>(1)</sup>	PTFE <sup>(2)</sup>	PA66 <sup>(3)</sup>
Density [g/cm <sup>3</sup> ]	1.31	2.15	1.15
Young's modulus [MPa]	4200	22	3500
Tensile strength [MPa]	116	-	84
Elongation at break [%]	15	220	70
Hardness	253 <sup>(4)</sup>	55 <sup>(5)</sup>	175 <sup>(4)</sup>
Max. op. temperature [°C]	260	260	100

<sup>(1)</sup> Polyether ether ketone. <sup>(2)</sup> Polytetrafluoroethylene. <sup>(3)</sup> Polyamide 66. <sup>(4)</sup> Ball indentation hardness ISO 2039-1 [N/mm<sup>2</sup>]. <sup>(5)</sup> Shore D.

## 2.2. Ageing Process of the Different Materials

With reference to the ASTM D7216-15 standard [15], the ageing of the different materials was achieved by immersion of the specimens in four standard automatic transmission oils. A MEMMERT Universal Oven (Model U, precision: ± 0.5 °C) was used to maintain the temperature at 100 °C. The specimens were immersed in the oils for 168 h, as specified in the standard. In addition, specimens of each material were aged for 336, 504 and 672 h to verify the compatibility of the different polymer/oil combinations for longer periods.

Before starting the aging period, the specimens were cleaned with a moist cloth so that no remains or particles from the process of material machining remained on their surfaces. They were then submerged in ethanol and dried with blotting paper and hot air. After extraction from the oven, the specimens were cleaned by placing them on blotting paper to drain the oil. Ethanol was used to ensure complete cleaning, and excess ethanol was removed with blotting paper to avoid its evaporation on the surface of the specimens.

## 2.3. Materials Characterisation

The evaluation of the compatibility of the three abovementioned materials with the four ATFs was based on the ASTM-D7216 standard, which determines the processes for testing changes in volume, tensile strength, and hardness. All of these properties were measured before and after the ageing of the materials in the ATFs.

Changes in volume were determined using a Radwag AS 310.R2 Analytical Balance (readability: 0.1 mg). A recipient full of water was weighed in the balance with the specimens hanging from a hook, and thus not adding their weight to that of the water but displacing a certain amount of water corresponding to the volume of the specimen. The volume before and after the ageing period was calculated by the difference in weight. The hardness of three specimens of each material was measured with a SAUTER HBD 100 durometer before the ageing process to be taken as a reference. Later, the hardness of each aged specimen was measured at four different points. The mean value of these four measurements was considered to be the result to be compared with the reference value. The

tensile strength was measured with an MTS© Sinergy traction machine with a charge cell of 5 kN (MTS-166). The traction speed was set depending on the material and immersion time (Table 4), according to the recommendations of the ASTM D6838 to keep the testing time below 5 min.

**Table 4.** Traction speed (in mm/s) by material and ageing time.

Material	Ageing Time (h)			
	168	336	504	672
PEEK	1.5	1.5	1.5	1.5
PTFE	20	20	20	18
PA66	3	3	3	3

#### 2.4. Complementary Tests

The X-ray diffraction (XRD) experiments were performed to calculate the degree of crystallinity of the polymers before and after ageing in the different oils. The XRD diagrams were collected with Cu radiation ( $K_{\alpha 1}$ ,  $K_{\alpha 2}$ ), using a PHILIPS X'PERT PRO diffractometer in a  $2\theta$  range of  $5\text{--}80^\circ$ , with a step size of  $0.0167^\circ$ .

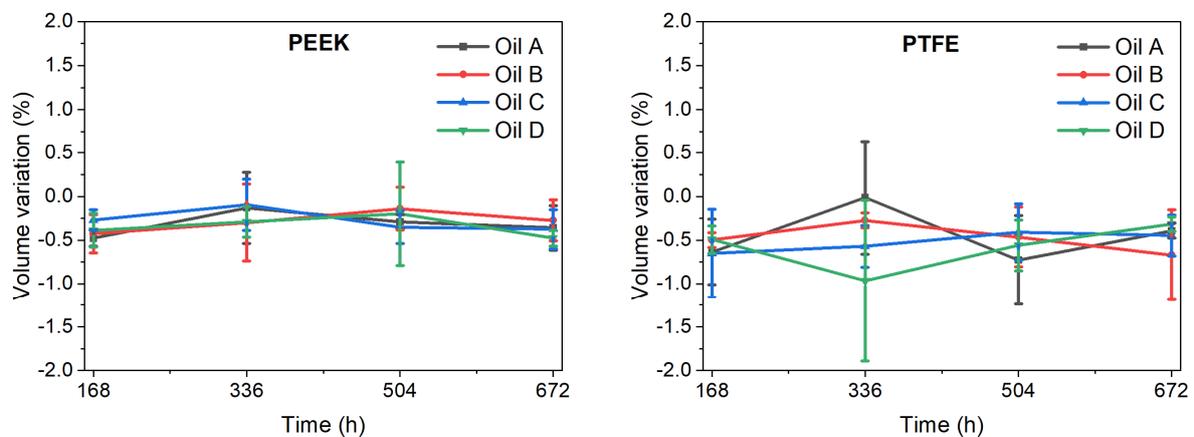
Fourier transformed infrared spectroscopy (FTIR) measurements were performed on the polymers in a Varian 670-IR FTIR spectrometer with a Golden Gate ATR device to check possible variations in their composition after the ageing process. The spectrometer resolution range was from 4 to  $2000\text{ cm}^{-1}$  and 16 scans were taken for each sample.

Differential scanning calorimetry (DSC) tests were also performed on the polymers to check possible changes in their crystalline structure. The tests were performed between 100 and  $400\text{ }^\circ\text{C}$ , with a heating speed of  $20^\circ\text{ C/min}$  in an inert atmosphere ( $\text{N}_2\text{--}100\text{ mL/min}$ ) and using a platinum crucible. The equipment was an SDT Q600 from TA Instruments.

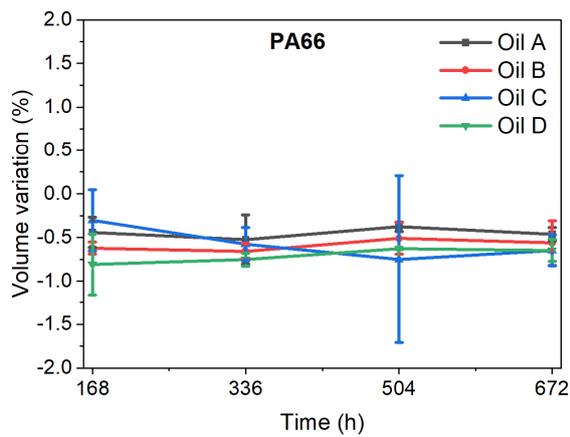
### 3. Results and Discussion

#### 3.1. Changes in Volume

The compatibility of the polymers with the ATFs with respect to volume change was tested initially for the time (168 h) specified in the ASTM D7216-15 standard [15]. In general, changes in volume depended more on the material type than on the oil used (Figure 1). The three structural materials (PEEK, PTFE, and PA66) experienced a slight reduction in volume. Although there are no specifications for the compatibility of these materials with ATFs, they seem to be quite reliable when in contact with these oils, according to the measured volume variations, which are around  $-0.5\%$ .



**Figure 1.** Cont.

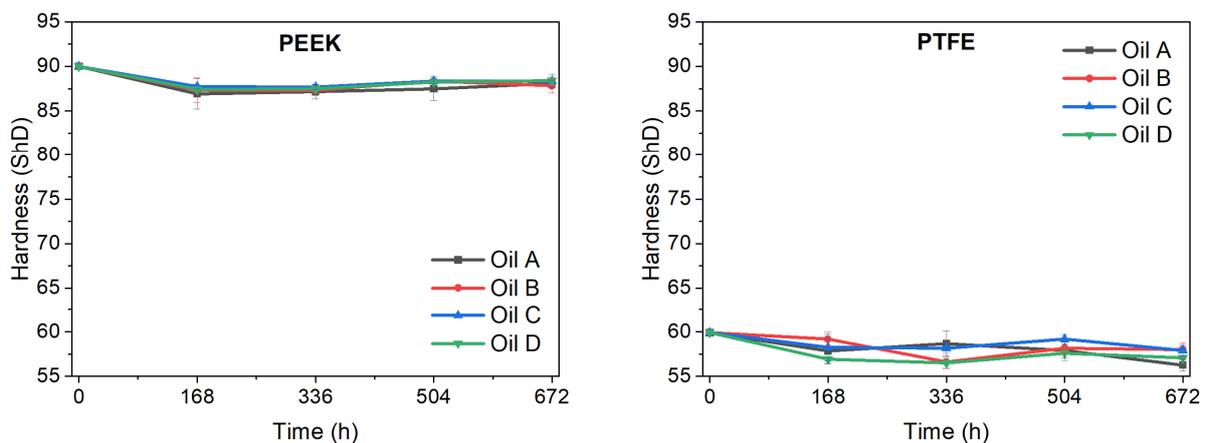


**Figure 1.** Changes in volume of PEEK, PA66, and PTFE after immersion in oils A, B, C and D.

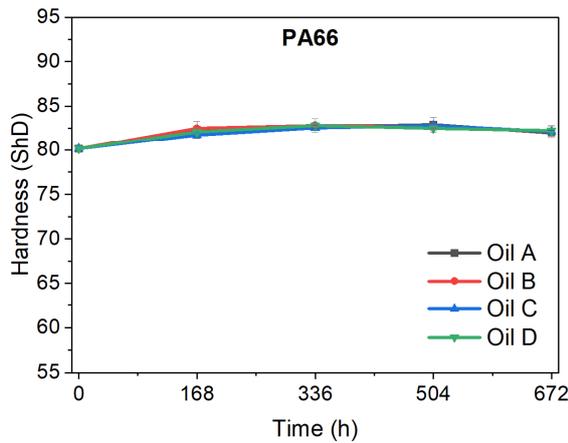
After longer ageing times (from 168 h upwards), PEEK and PA66 hardly changed in volume. The volume variations remained, in general, between 0 and −1%. On the other hand, PTFE showed greater volume variations than its counterparts. Although there are no specifications for these materials when aged in ATFs regarding the admissible changes in volume, it could be concluded that all the materials underwent negligible volume changes. These changes were negligible, not only during the time established in the standardised test, but also for longer time periods.

### 3.2. Changes in Hardness

In a similar way to the volume variation tests, there are no specifications for changes in the hardness of polymers aged in ATFs. In general, only small changes in hardness were measured after the first 168 h in the three tested materials (Figure 2). Since the hardness of PEEK and PTFE decreased by around 3 points (Shore D), PA66 was the only material that hardened a little after ageing. The changes in hardness of every material were similar, irrespective of the oil used. From 168 h upwards, the materials showed negligible variation in hardness, which leads us to the conclusion that they are compatible with the ATFs in the tests.



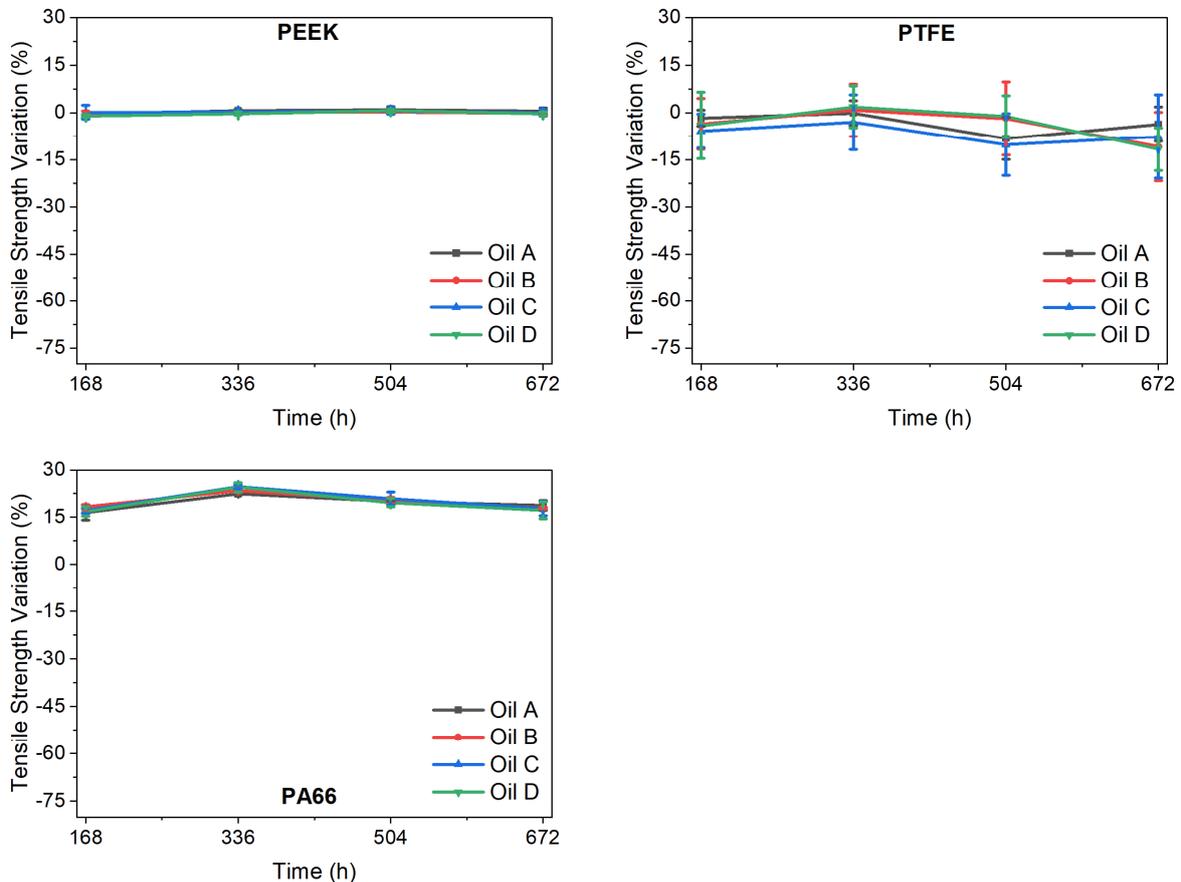
**Figure 2. Cont.**



**Figure 2.** Changes in hardness of PEEK, PTFE, and PA66 (Shore D) after immersion in the ATFs.

**3.3. Changes in Tensile Strength and Elongation at Break**

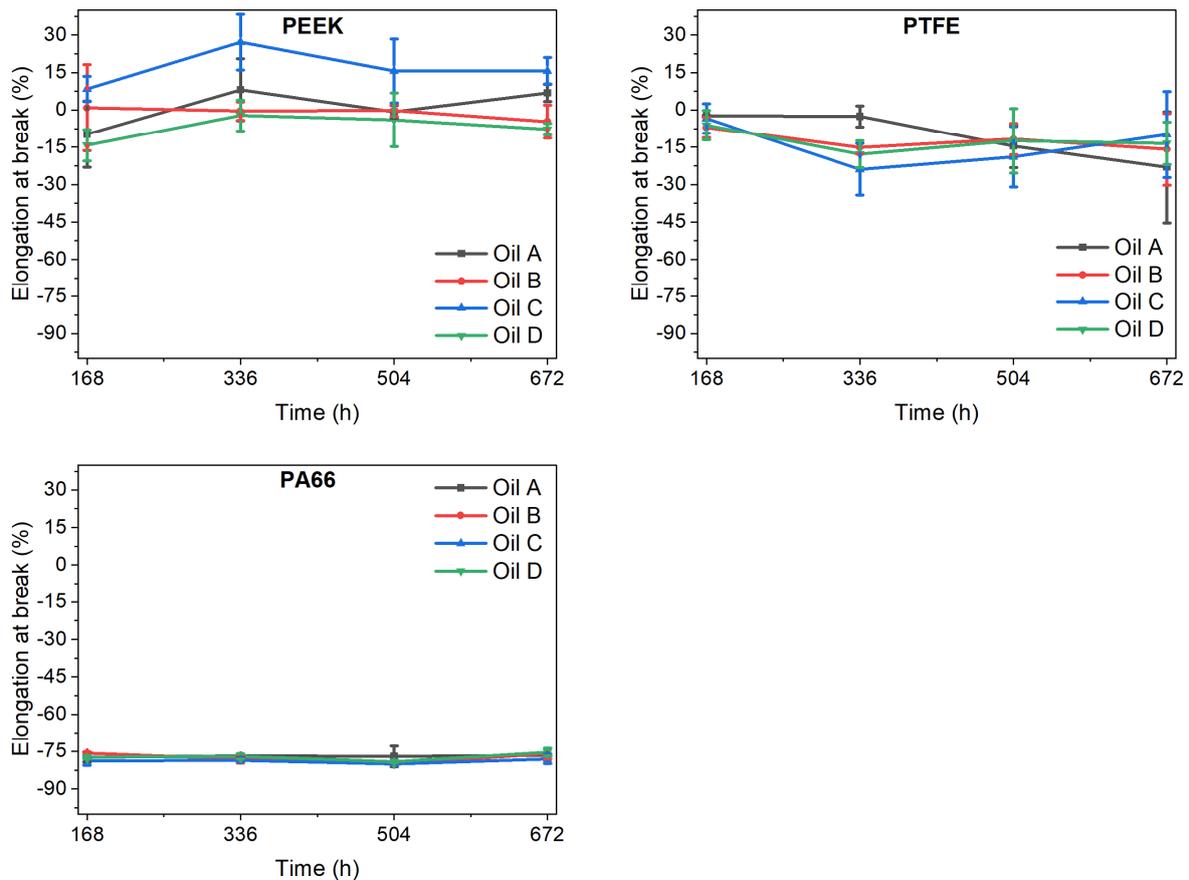
The stress–strain curves of all materials were obtained before and after their ageing in the ATFs for different periods of time (see Supplementary Information, Figures S1–S3), and the tensile strength and the elongation at break were obtained from these curves. The polymers had two types of tensile strength behaviour at an aging time of 168 h (Figure 3), depending on the ATF used. On one hand, the PEEK and PA66 tensile strength variation did not depend on the oil used. On the other hand, PTFE showed mean tensile strength variations of between  $-1.5\%$  (oil A) and  $-6\%$  (oil C). Nevertheless, while the tensile strength of PEEK remained constant, regardless of the oil and the ageing time used, this property increased slightly for PA66 and decreased slightly for PTFE.



**Figure 3.** Changes in tensile strength of PEEK, PTFE, and PA66 after ageing in the ATFs.

From 168 h upwards, all materials maintained relatively constant tensile strength values. PEEK and PTFE seem to be compatible with these ATFs, showing low tensile strength variations (null in the case of PEEK), but PA66 should be studied further.

The elongation at break was more affected by the ageing process than was the tensile strength (Figure 4). In the case of PEEK, variations in elongation at break depended on the ATF used and were between +7% and −15% at 168 h. On the contrary, the PTFE results are aligned with those of tensile strength, with variations in the elongation at break values of between −2 and −7%. Once again, PA66 had contrasting results, with a reduction in elongation at break of around 77%, regardless of the ATF used. This is a clear sign of incompatibility, as the material became less flexible, and thus more fragile.



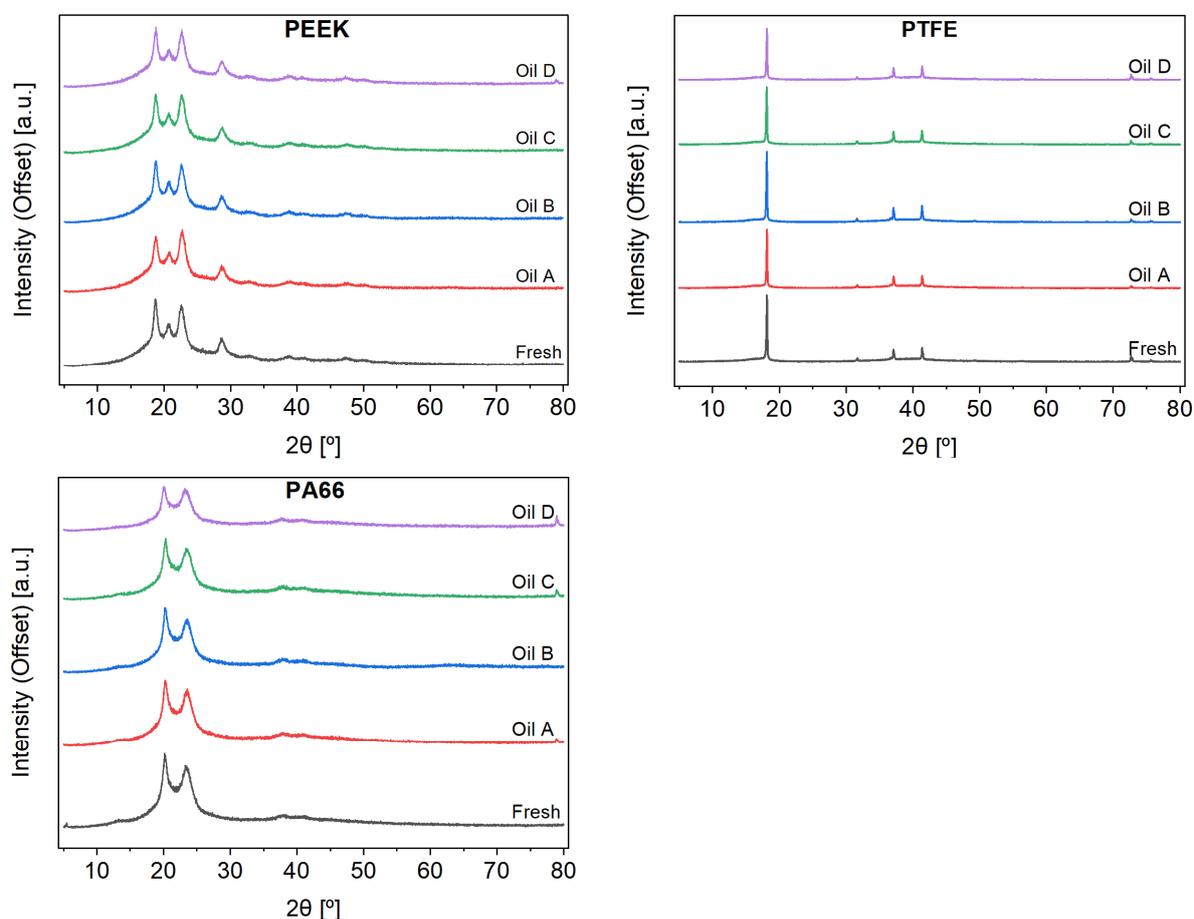
**Figure 4.** Changes in elongation at break of, PEEK, PA66, and PTFE after immersion in the ATFs.

From 168 h upwards, elongation at break seemed to be more affected by immersion in the ATFs than was the tensile strength, especially in the cases of PEEK and PTFE. PA66 was the only material showing similar elongation at break regardless of the ageing time and oil used, although it showed the greatest reduction in this property. Lann et al. [13] and Decker et al. [14] pointed out the tendency of PA66 to crystallize when aged in ATFs, although they concluded that it was the high temperature that caused this effect. According to Figures 3 and 4 and Figure S3, this embrittlement was independent of the type of oil, although in this case the tensile strength increased (15%). On the other hand, PTFE and PEEK showed lower variation in elongation at break than the PA66 and a higher dependency on the oil used.

### 3.4. Complementary Tests

Complementary tests (XRD, FT-IR, and DSC) were performed on the polymers. X-ray diffraction (XRD) experiments were used to search for changes in the crystalline structure of the materials. However, the XRD spectra (Figure 5) showed no noticeable changes in the

crystalline/amorphous areas ratio, which means that it is not possible to correlate these results with the reported changes in the mechanical properties.



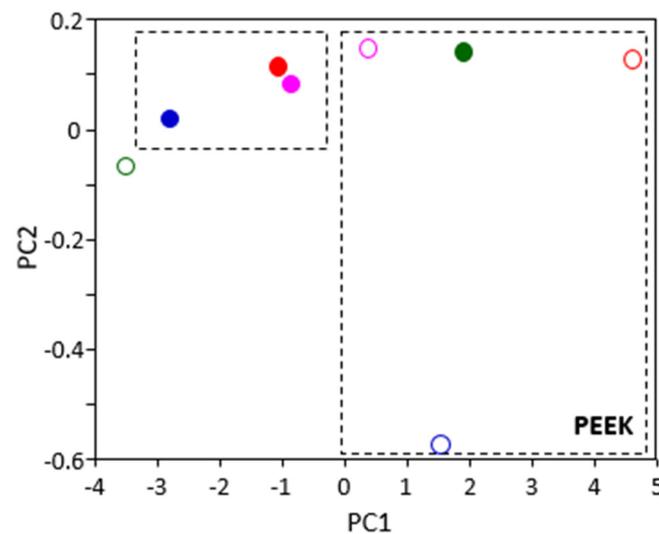
**Figure 5.** XRD spectra for the polymers (fresh, after 168 h, and after 672 h) in the ATFs.

Infrared spectroscopy (FTIR) was used to detect possible changes in the polymeric structure of the materials. In the FTIR spectra, the qualitative differences (mainly peak positions) in the spectra of the same material subjected to different ageing conditions are almost negligible. Nevertheless, possible quantitative differences were evaluated by comparing peak areas. Tables S1–S3 record the peak areas of PEEK, PTFE, and PA66 when fresh and under different ageing conditions. The area ratio of aged material vs. fresh material is calculated for every sample at every peak. In principle, this ratio should be similar for all the peaks within a sample. We considered deviations to be significant in those samples with at least one area acting as outlier according to a two-sided Grubb's test ( $p = 0.05$ ) [16]. With this criterion, PTFE and PA66 did not show significant differences. However, PEEK in oil C (at both tested times) showed an increase in the absorbances at  $2850\text{ cm}^{-1}$  and  $2915\text{ cm}^{-1}$ , attributable to  $-\text{CH}-$  stretching vibrations, suggesting a particular influence of this oil. The sample aged in oil B for 672 h showed a similar trend, whereas the sample aged in oil A for 168 h showed a decrease in absorbance at  $2915\text{ cm}^{-1}$ .

Principal component analysis is a data analysis methodology that allows the conversion of a group of correlated variables (in this case, IR absorbance at different wavenumbers) into a smaller number of uncorrelated variables which, in addition, retain most of the variability of the original data [17].

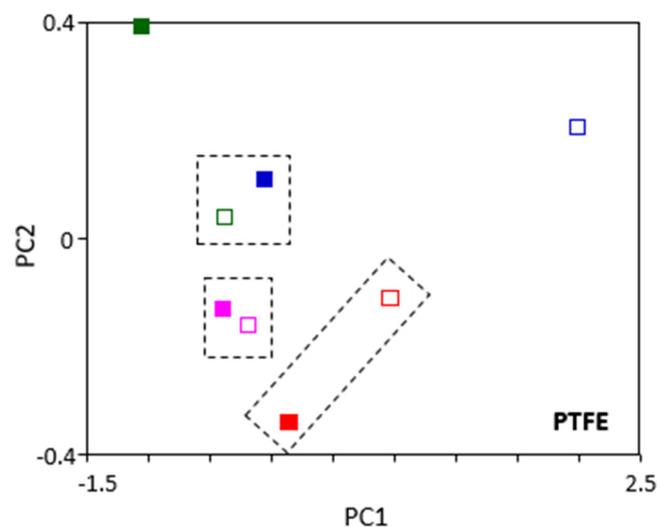
According to PCA analysis of the spectra of the different polymers, the samples of PEEK with 168 h of ageing (solid markers) group together (Figure 6), as do the samples with 672 h of ageing (empty markers), except for oil B (green markers). These results suggest

that, in the case of PEEK, time causes greater effects the oil type does. This would explain, in general, the similarity of results in the PEEK mechanical tests after ageing in the ATFs.



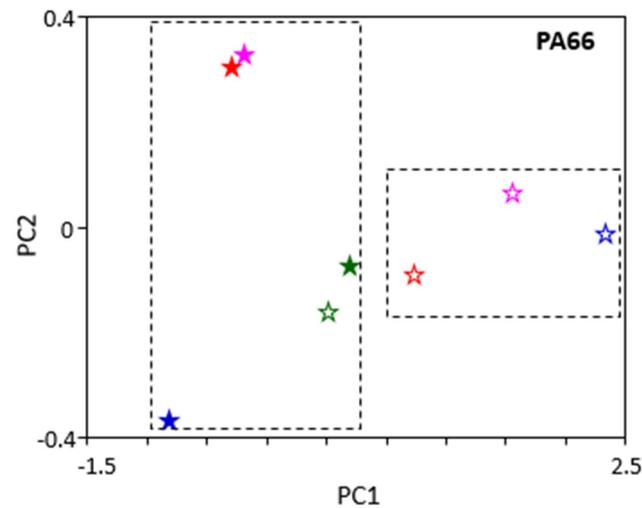
**Figure 6.** PCA analysis of the FT-IR spectra of the PEEK (oil A (blue), oil B (green), oil C (fuchsia), oil D (red), 672 h (empty markers) and 168 h (solid markers)).

However, the PCA analysis of aged PTFE samples (Figure 7) revealed the following three groups: oil C (fuchsia markers) after 168 and 672 h, oil D (red markers) after 168 and 672 h, and oil A (blue markers) after 672 h and oil B after 168 h. According to these findings, in the case of oils C and D, the oil type is more important than the ageing time. This is well represented in the dispersion of results for the mechanical properties variation tests, depending on the ATF used in the PTFE ageing.



**Figure 7.** PCA analysis of the FT-IR spectra of the PTFE (oil A (blue), oil B (green), oil C (fuchsia), oil D (red), 672 h (empty markers) and 168 h (solid markers)).

PA66 showed similar behaviour to that of PEEK (Figure 8). Samples with shorter ageing times grouped together, as did samples with longer ageing times, except with oil B (green markers). This suggests, again, that the ageing time is more important for the PA66 samples than the oil type. These would explain the similarity of results in the PA66 mechanical tests after ageing in the different ATFs.



**Figure 8.** PCA analysis of the FT-IR spectra of the PA66 (oil A (blue), oil B (green), oil C (fuchsia), oil D (red), 672 h (empty markers) and 168 h (solid markers)).

Differential scanning calorimetry (DSC) was used to check if there were changes in the fusion processes of the materials and, thus, in their crystalline structure. The DSC results for the different material/oil combinations are shown in Figure S7, and Tables 5–7 were obtained from those curves.

**Table 5.** DSC results for the PEEK samples (fresh and aged in the ATFs).

Sample	$\Delta H$ [J/G]	$T_{PEAK}$ [°C]	$T_{ONSET}$ [°C]
Fresh	31.60	338	315
Oil A	48.50	336	321
Oil B	76.16	337	313
Oil C	73.37	337	307
Oil D	45.89	339	318

**Table 6.** DSC results for the PTFE samples (fresh and aged in the ATFs).

Sample	$\Delta H$ [J/G]	$T_{PEAK}$ [°C]	$T_{ONSET}$ [°C]
Fresh	39.18	336	324
Oil A	41.28	336	324
Oil B	40.14	338	324
Oil C	41.30	336	324
Oil D	42.95	335	325

**Table 7.** DSC results for the PA66 samples (fresh and aged in the ATFs).

Sample	$\Delta H$ [J/G]	$T_{PEAK}$ [°C]	$T_{ONSET}$ [°C]
Fresh	11.03	258	250
Oil A	19.30	258	248
Oil B	17.43	257	246
Oil C	29.71	260	249
Oil D	21.26	258	249

The enthalpy of fusion ( $\Delta H$ ) for a fully crystalline PEEK would be 164.85 J/g [18], so the fresh sample, with 31.60 J/g, was not crystalline. When the PEEK was aged in the different ATFs, a rise in the enthalpy associated with an increase in crystallinity was detected (Table 5), especially for those samples aged in oils B and C. However, this change in the crystalline structure was not large enough to modify the mechanical characteristics

of the PEEK. The  $T_{\text{Onset}}$  and  $T_{\text{Peak}}$ , represent the start of the fusion process and the fusion temperatures, respectively. PEEK is the material that shows the greatest difference between them, and the highest values of all the materials, although very similar to those of PTFE. The difference between  $T_{\text{Onset}}$  and  $T_{\text{Peak}}$  might be associated with crystallization taking place during the heating process, with the possibility of the peak being double, depending on the heating speed [18].

In the case of PTFE, the  $\Delta H$  at fusion for a crystalline sample would be 57.3 J/g [19,20]. The results therefore show that, as in the case of PEEK, the fresh sample is not crystalline (Table 6). The slight increase in enthalpy after ageing does not suggest a notable change in the crystalline structure, confirming the results of the mechanical tests, with very small changes being seen.

Finally, Table 7 shows the DSC results for PA66. This material showed the greatest changes in mechanical properties (+7 ShD points, +15% tensile strength, and –80% elongation at break) of all the materials in this study. These variations would suggest a certain degree of crystallization, which has been mentioned by Lann et al. [13] and Decker et al. [14] after an ageing process of PA66 in ATFs. PA66 is a semi-crystalline polymer [21–23], and the variation in the degree of crystallinity can be observed in the evolution of the melting enthalpy [24]. Table 7 shows that the melting enthalpy nearly doubles after ageing in the different ATFs, meaning that the increase in the degree of crystallinity is proportionately greater than in the other studied polymers. Although the amorphous phase is still more prevalent, according to the XRD results, the change in the structure of PA66 is enough to cause variations in its mechanical properties.

Finally, according to the changes in the different mechanical properties tested for the materials after being aged in the four ATFs, PEEK and PTFE may be considered to be compatible in all cases, as the changes in volume, hardness and tensile strength are negligible. Although they presented higher variations in their elongation at break, we consider these variations to be acceptable. That is not the case for PA66, which showed a very large decrease (~75%) in elongation at break. This, together with the increase in tensile strength (15%) and a slight increase in hardness (the hardness of the other polymers decreased), denotes a certain embrittlement of the PA66. These results allow the degree of compatibility of the ATFs tested with these polymers to be established, as shown in Table 8.

**Table 8.** Oil compatibility with the polymers studied.

ATFs	Polymers		
	PEEK	PTFE	PA66
Oil A	***	***	**
Oil B	***	***	**
Oil C	***	***	**
Oil D	***	***	**

\*\* Medium compatibility; \*\*\* High compatibility.

#### 4. Conclusions

Four conventional ATFs were tested for their compatibility with three polymeric materials that can be used in electrified transmissions. After testing volume, hardness, tensile strength and elongation at break, and measuring variation in these materials when aged in the ATFs, and additionally after some complementary tests to justify the results, we can conclude that:

- PEEK and PTFE showed full compatibility in the short and long-term regarding volume, hardness, and tensile strength properties with all the ATFs used. PEEK showed good compatibility (lower variations) with all the ATFs for elongation at break also, while PTFE showed worse results than PEEK on this measure.

- PA66 had worse compatibility with the oils than the other materials. This result was related to an increase in the grade of crystallinity of the PA66 that caused changes in the mechanical properties of the aged samples.
- According to the FTIR results, the variations in the mechanical properties of the materials are, in general, related more to the ageing time than to the oil type, with the exception of the PTFE, which does not seem to be much affected by the ageing time.
- The ATFs showed good compatibility with the studied polymers and only PA66 showed worsening of its elongation at break performance after ageing.
- The compatibility of these ATFs with the tested materials is a necessary condition, but is not in itself sufficient, to allow their use in electrified transmissions. Additional oil compatibility tests (electrical, magnetic, thermal, corrosion, aeration, and foaming at high speeds) must be performed to justify their use in electrified transmissions. In addition, the results of these acceptance tests should not be used for predicting the lifetime of a component in a real application.

**Supplementary Materials:** The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/app12073608/s1>, Figures S1–S3: Tensile strength and elongation and break of the PEEK, PTFE and PA 66 (fresh and after 168, 336, 504 and 672 h of ageing in the ATFs); Figures S4–S6: FT-IR spectra of the PEEK, PTFE and PA 66 (fresh and after 168 and 672 h of ageing in the ATFs); Figure S7: DSC results for the different material/oil combinations (Exo-up); Tables S1–S3: Grubb’s statistic for every peak ratio for PEEK, PTFE and PA 66.

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